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# *Indian Standard*

## SPECIFICATION FOR *AVARAM* BARK

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# Indian Standard

## SPECIFICATION FOR AVARAM BARK

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# Indian Standard

## SPECIFICATION FOR *AVARAM* BARK

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 17 April 1969, after the draft finalized by the Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

**0.2** *AVARAM*, a shrub, (*Cassia auriculata* Linn., fam. Leguminosæ) also known as *TARWAD*, *TARWAR*, *AVAL*, *TANGEDU* or *AWAL* is a source of one of the best vegetable tanning materials available indigenously. It occurs in abundance in some parts of the country, particularly in Madras, Andhra Pradesh, Maharashtra, Mysore and Rajasthan. Its bark is extensively used in Madras and Bombay tanneries for the production of East India tanned leather.

**0.3** The bark is collected by cutting the shoots or branches of the shrub and stripping by beating the sticks with a stone. The peeled bark is then dried under shade and cut into pieces of about 25 mm in width and 100 to 150 mm in length. The dry bark has a light brown or cinnamon colour.

**0.4** The infusion of *AVARAM* bark is self-bating and penetrates rapidly, yielding a pale-coloured tough and elastic leather. Used alone, the leather may develop a reddish colour when exposed to light and air, but this is prevented when the tanning is completed with myrobalan infusion.

**0.5** The methods of test are based on the 'Official methods of analysis' published by the Society of Leather Trades' Chemists, U. K. However, to suit Indian conditions the temperature of cooling the infusion before testing has been changed from 18° to 27°  $\pm$  2°C as this does not adversely affect the repeatability and reproducibility of the method.

**0.6** This standard contains clause 4.1 which calls for agreement between the purchaser and the supplier.

**0.7** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained

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\*Rules for rounding off numerical values (revised).

in the rounded off value should be the same as that of the specified value in this standard.

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## 1. SCOPE

**1.1** This standard prescribes the requirements and the methods of sampling and test for *AVARAM* bark intended for tanning.

## 2. TERMINOLOGY

**2.1** For the purpose of this standard, the definitions given in IS : 1640-1960\*, shall apply.

## 3. REQUIREMENTS

**3.1 Material** — The material is the bark of the species *Cassia auriculata* Linn., fam. Leguminosæ, commonly known as *AVARAM*, cut into pieces and dried.

**3.2** The material shall comply with the requirements given in Table 1, when tested according to the methods mentioned against each of the characteristic.

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**TABLE 1 REQUIREMENTS OF AVARAM BARK**

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST ( REF TO CL NO. IN APPENDIX A )
(1)	(2)	(3)	(4)
i)	*Tannins, percent by weight, <i>Min</i>	15	A-2
ii)	*Non-tannins, percent by weight, <i>Max</i>	12	A-2
iii)	Moisture, percent by weight, <i>Max</i>	12	A-3
iv)	pH of analytical strength solution, <i>Min</i>	4.7	A-4
v)	Colour: Yellow/red Red, <i>Max</i>	1.5 5.5	A-5

\*Calculated on moisture-free basis.

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## 4. PACKING AND MARKING

**4.1 Packing** — Unless otherwise agreed to between the purchaser and the supplier, the *AVARAM* bark shall be packed in fairly light-proof packages like gunny bags.

\*Glossary of terms relating to hides, skins and leather.

**4.2 Marking** — The containers shall be marked on the outside with the following information:

- a) Name of the material;
- b) Net weight of the material;
- c) Supplier's name and recognized trade-mark, if any; and
- d) Date of packing.

**4.2.1** The containers may also be marked with the ISI Certification Mark.

**NOTE** — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## 5. SAMPLING

**5.1** The scale of sampling and criteria for conformity of the material to the standard shall be as prescribed in Appendix B.

## A P P E N D I X   A

*( Table 1 )*

### METHOD OF TEST FOR AVARAM BARK

#### A-1. QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals shall be employed, and distilled water ( conforming to IS : 1070-1960\* ) shall be used where the use of water as a reagent is intended.

**NOTE** — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

#### A-2. DETERMINATION OF TANNINS

**A-2.0 Principle** — The tanning matter absorbable by hide powder is determined by finding the difference between the percentages of total solubles and the non-tannins.

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\*Specification for water, distilled quality (*revised*).

### A-2.1 Apparatus

A-2.1.1 *Glass Cylinder* — 500 ml capacity.

A-2.1.2 *Berkefeld Candle* — one.

A-2.1.3 *Filtration Flask* — 500 ml capacity.

A-2.1.4 *Porcelain Dish* — flat-bottom, capacity 45 ml, conforming to IS : 2837-1964\*.

A-2.1.5 *Filtration Apparatus* — as shown in Fig. 1.

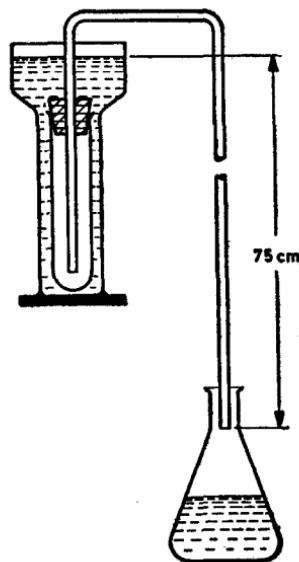


FIG. 1 FILTRATION APPARATUS

### A-2.1.6 *Steam-Bath*

A-2.1.7 *Vacuum Oven* — with thermostatic control, capable of maintaining  $100^{\circ} \pm 2^{\circ}\text{C}$ .

### A-2.2 Reagents

A-2.2.1 *Hydrochloric Acid* — conforming to IS : 265-1962†.

\*Specification for porcelain crucibles and basins.

†Specification for hydrochloric acid (revised).

**A-2.2.2 Chrome Alum Solution**— Dissolve 30 g of chrome alum conforming to IS : 332-1951\* in distilled water and make up the solution to one litre. Chrome alum solution more than 30 days old shall not be used.

**A-2.2.3 Kaolin**— Kaolin shall be of such quality that when 1 g of it is suspended in 100 ml of distilled water and well shaken, the *pH* of the suspension shall be between 4·0 and 6·0, that is, it shall neither give red colour with methyl orange nor deep purple colour with bromocresol purple. When 1 g of kaolin is shaken with 100 ml of 0·01 N acetic acid and the mixture filtered, the filtrate shall leave less than 1 mg of residue after evaporation and drying.

**A-2.2.4 Chromed Hide Powder**— For each analysis digest a multiple of the quantity of hide power (see A-2.2.6) containing 6·25 g of dry matter, with ten times its weight of distilled water for one hour. Add to this 1 ml of chrome alum solution for each gram of air-dry hide powder taken, stir frequently for several hours and then allow to stand overnight. Transfer the chromed powder to a clean linen (filter cloth), drain and squeeze. Place the cloth containing the powder in a suitable vessel (an enamel bucket is suitable for large quantities), open out the cloth bag and pour on to the powder a quantity of water equal to 15 times the weight of the air-dry hide powder taken. Mix the powder and water thoroughly and digest for 15 minutes, lift out the cloth and powder, and immediately drain and squeeze the powder so that it contains approximately 75 percent moisture. Digest the powder three more times in the same way in distilled water. At the end of final digestion thoroughly break up the cake of chromed powder, mix uniformly and free from lumps and weigh the whole.

**A-2.2.5 Linen**— Pieces of linen cloth shall be used for washing the chromed hide powder and for the preliminary filtration of detannized solutions. The linen shall be freed from weighting matter by boiling in several changes of distilled water.

**A-2.2.6 Hide Powder**— It shall comply with the following requirements:

- The ash content of the powder shall be not more than 0·3 percent, by weight; and
- When 7 g of the air-dry powder is allowed to stand in contact for 24 hours with 100 ml of 0·1 N potassium chloride solution, previously adjusted to *pH* 5·5 by addition of 0·01 N acetic acid, then the liquor obtained by centrifuging or by filtering through a paper previously washed with the above potassium chloride solution shall have a *pH* ranging between 5·0 and 5·5.

\*Specification for chrome alum, potash.

**A-2.2.7 Gelatin-Salt Reagent** — Dissolve 1 g of photographic grade gelatin and 10 g of sodium chloride in 100 ml of distilled water at a temperature not higher than 60°C and adjust the pH to 4.7 approximately by adding acid or alkali, that is, the solution shall give red colour with methyl red and yellow with methyl orange. To preserve this solution, add 2 ml toluene ( conforming to IS : 1839-1960\* ) and keep in a cool place. Freshly prepared solution is preferable.

**A-2.3 Procedure** — This comprises firstly in the determination of total solubles and secondly of non-tannins.

**A-2.3.1 Determination of Total Solubles**

**A-2.3.1.1 Preparation of a solution** — Weigh out such a quantity of material as will give nearly as possible 4.0 g per litre of tanning matter absorbable by hide powder, and in any case not less than 3.75 g and not more than 4.25 g.

**NOTE** — In the event of the results of an analysis showing a tanning strength outside these limits, the analysis shall be repeated employing the required amount of material.

The material shall be ground suitably until it passes through a 1.40-mm IS Sieve, then sieved through a 600-micron IS Sieve. If it cannot be ground so as to pass entirely through the specified sieves the finer and coarser portion shall be separately weighed so as to determine the proportions of fibre, fine and coarse material in the whole amount ground. The quantity of material actually used for extraction shall consist of fibre, fine and coarse material in the same proportions.

**NOTE** — The apertures of BS sieve 12, ASTM sieve 14 ( also known as 1.41 mm US standard sieve ) and BS sieve 25, ASTM sieve 30 ( also known as 595-microns US standard sieve ) are within the limits laid down for 1.40 mm and 600-microns IS sieves respectively, and may, therefore, be used in their place.

Take such a quantity of material which will give as nearly as possible 2 litres of solution of required analytical strength by using *Proctor Extractor* described below:

**Proctor Extractor** — An assembly of the apparatus is given in Fig. 2. Essentially it consists of a tall shape beaker without spout, having a capacity of about 250 ml. The beaker shall be kept in a water-bath. Cover the bottom of the beaker to a height of 20 mm with sand, washed with hydrochloric acid, then with water and finally dried. Bend a thistle funnel twice at right angles. Cover the bell of the funnel ( about 35 mm in diameter ) with a piece of well-washed muslin and insert it in the layer of sand in the beaker. Attach a rubber tubing to the stem at the other end of which is a

\*Specification for toluene, reagent grade.

glass tube ending inside a collecting flask. A conical flask of capacity 1 000 ml, shall be kept in the water-bath alongside the beaker. The tanning material to be extracted is put into the beaker without disturbing the sand layer and covered with distilled water. The further supply of water for extraction shall come from the conical flask. By regulation of the stop-cock and the screw clip, the inflow and outflow shall be so adjusted that the liquor in the beaker always stands at the same height.

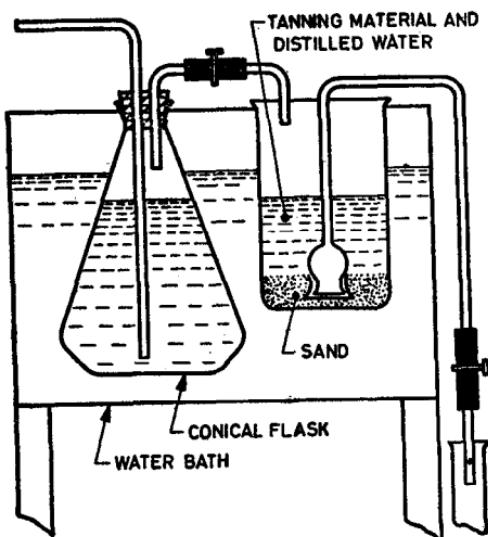


FIG. 2 PROCTOR EXTRACTOR

The ground material shall be soaked in cold distilled water in the extractor for not less than 12 and not more than 18 hours (for example overnight, before commencing extraction ).

At the end of this time draw off the infusion and continue the extraction at such uniform speed that the required 2 litres shall be obtained in 4 hours. After collecting the first 150 ml raise the temperature of the water-bath to 50°C and at this temperature, collect an additional 750 ml. Then raise the temperature rapidly to boiling and extract the further quantity required to make 2 litres as near boiling as possible.

Immerse the flask in a large vessel ( a laboratory sink is convenient ) containing water at  $27^{\circ}\pm 2^{\circ}\text{C}$  throughout the cooling process, agitating the flask from time to time. Continue the cooling until the temperature of the contents of the flask has reached  $27^{\circ}\pm 2^{\circ}\text{C}$ . Make up to the mark with water kept at the same temperature.

NOTE 1 — It is essential that at no time during the cooling shall the temperature of the water in the cooling vessel fall below  $27^{\circ}\pm 2^{\circ}\text{C}$  in order to avoid local cooling at the walls of the flask.

NOTE 2 — In hot climates, where it is difficult to maintain the temperature, it is recommended that the flasks, after cooling, be placed in paper bags.

**A-2.3.1.2 Procedure** — Pour about 400 ml solution (as prepared in A-2.3.1.1) into a glass cylinder into which dips a clean and dry filter candle. Leave the candle immersed in tannin solution for about 10 minutes before starting the filtration. The necessary suction may be obtained by using a column of solution of 75 cm length (see Fig. 1) or by using a suction pump keeping the vacuum fairly constant never exceeding 20 cm of mercury. Reject the first 250 ml of the filtrate and continue the filtration until it is clear to both transmitted and reflected light. Evaporate the next 50 ml of the filtrate in a porcelain dish to dryness and weigh. Repeat the process of drying and weighing until constant weight is obtained. If a clear filtrate cannot be obtained by the use of the candle, then mix 1 g of the kaolin per 250 ml of the solution before filtration. If the tanning solution is optically clear in the unfiltered state it need not be filtered.

NOTE — In case Barkefeld candles are not available, the following procedure shall be adopted for filtration through fluted filter paper\*.

To 1 g kaolin in a beaker add sufficient solution to fill the filter paper, stir and pour on a filter paper. Return filtrate to paper when approximately 25 ml has been collected, repeating operation for one hour, being careful to transfer all kaolin to the paper. At the end of one hour remove solution from filter paper, disturbing the kaolin as little as possible. Refill the paper with original solution and begin to collect the filtrate in a fresh beaker as soon as it comes optically clear. The paper shall be kept full and the funnels and collecting vessels shall also be covered at the time of filtration. Pipette 50 ml of the filtrate at  $27^{\circ}\pm 2^{\circ}\text{C}$  in a porcelain basin; evaporate; dry and weigh. Repeat the process of drying and weighing until constant weight is obtained.

#### **A-2.3.1.3 Calculation**

$$\text{Total solubles, percent by weight} = \frac{40 W_1 \times 100}{W}$$

where

$W_1$  = weight of solubles in g from 50 ml of the filtrate, and  
 $W$  = weight in g of *AVARAM* bark taken.

#### **A-2.3.2 Determination of Non-Tannins**

\*Whatman No. 11/No. 2 or Munktells' No. 1F or equivalent is suitable.

**A-2.3.2.1 Procedure** — Weigh accurately a quantity of wet hide powder (say  $X$  g) containing about 6.25 g of dry powder (A-2.2.4) and add it immediately to 100 ml of the unfiltered tannin solution (prepared as in A-2.3.1.1) plus  $(26.25 - X)$  ml of distilled water already present in a shake-bottle of 150 to 300 ml capacity. Stopper the shake-bottle tightly with a rubber bung and mechanical rotary shaker and shake for exactly 10 minutes at 50 to 65 revolutions per minute. Pour powder and solution on a clean, dry filter cloth supported by a funnel, drain and squeeze by hand. Add to the filtrate 1 g of kaolin and pour into a 15 cm pleated filter paper\* returning the filtrate repeatedly until it is clear. Keep the funnel and collecting vessel covered during filtration. Test the filtrate with gelatin-salt reagent and if 10 ml gives any turbidity with 1 or 2 drops of the reagent, mention in the report of the analysis. Evaporate in a tared porcelain dish 50 ml of the filtrate and dry the residue in a vacuum oven at  $100^\circ \pm 2^\circ\text{C}$ ; cool and weigh until constant weight is obtained.

NOTE — To correct for the 20 ml of water of dilution introduced by the wet hide powder into the 100 ml of tannin solution the residue weight should be multiplied by 1.2. This corrected weight is the residue from 50 ml of original analytical solution.

### **A-2.3.2.2 Calculation**

$$\text{Non-tannins, percent by weight} = \frac{40 \times 1.2 \times W_1 \times 100}{W}$$

where

$W_1$  = weight of non-tannin in g from 50 ml of the filtrate after treatment with hide powder, and

$W$  = weight in g of the tanning material taken for the test.

## **A-2.4 Calculation of Tannin Content**

Tannins percent by weight = Percent total solubles in g (as obtained in A-2.3.1.3) — Percent non-tannins in g (as obtained in A-2.3.2.2).

NOTE 1 — All analysis should be done in duplicate. The absolute error in tannin content in duplicate analysis should not be more than 2 percent.

NOTE 2 — Where analysis are carried out by different chemists on the same sample, the result should not differ by more than 3 percent of the total tannin content.

## **A-3. DETERMINATION OF MOISTURE**

### **A-3.1 Apparatus**

#### **A-3.1.1 Wide-Mouth Weighing Bottle**

\*Whatman No. 11/No. 2 or Munketells' No. 1F or equivalent is suitable.

**A-3.1.2 Vacuum Oven** — with thermostatic control, capable of maintaining  $100^\circ \pm 2^\circ\text{C}$ .

**A-3.2 Procedure** — Transfer about 2 to 5 g of the ground material into a tared wide-mouth weighing bottle and weigh accurately. Dry it at about  $100^\circ \pm 2^\circ\text{C}$  in a vacuum oven for 3 to 4 hours, cool in a desiccator for about 20 minutes and weigh again accurately. Repeat the process of drying and weighing until two weighings at an interval of one hour do not differ by more than 2 mg.

### **A-3.3 Calculation**

$$\text{Moisture, percent by weight} = \frac{(W_1 - W_2) \times 100}{W_1}$$

where

$W_1$  = weight in g of the material taken for the test, and

$W_2$  = weight in g of the residue after drying.

## **A-4. DETERMINATION OF pH**

### **A-4.1 Apparatus**

**A-4.1.1 pH Meter** — with glass electrode.

**A-4.2 Procedure** — Determine the pH of the solution prepared in **A-2.3.1.1** after adjusting the relative density to 1.05 at  $27^\circ \pm 2^\circ\text{C}$  with cold water, by using a suitable pH meter.

## **A-5. DETERMINATION OF COLOUR**

### **A-5.1 Apparatus**

**A-5.1.1 Lovibond Tintometer** — having artificial light source\*.

**A-5.2 Procedure** — Fill the solution prepared in **A-2.3.1.1** immediately after filtration in 1-cm cell and determine the colour value. Calculate the result on 0.5 percent tannin content basis and express in units of red and yellow.

\*A 60-watt Osram Pearl lamp, or its equivalent is required, which should be changed after 200 hours' use.

# APPENDIX B

( Clause 5.1 )

## SAMPLING OF *AVARAM* BARK

### B-1. SCALE OF SAMPLING

**B-1.1 Lot** — In a single consignment all the *AVARAM* bark of the same form of cutting ( such as stick, chopped, ground, etc ) ground and dried under similar conditions shall constitute one lot.

**B-1.2** For ascertaining the conformity of the material to the requirements of this specification, each lot shall be considered separately. The number of packages to be selected for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

**TABLE 2 NUMBER OF PACKAGES TO BE SAMPLED**

No. of Packages in the Lot	No. of Packages to be Sampled
<i>N</i>	<i>n</i>
(1)	(2)
Up to 25	3
26 „ 50	4
51 „ 150	5
151 „ 300	6
301 „ 500	7
501 „ 1 000	8
1 001 and above	9

**B-1.3** The packages shall be selected at random from the lot. To ensure randomness of selection, use of random number tables ( see IS : 4905-1968\* ) shall be made. In case random number tables are not available, the following procedure may be adopted:

Starting from any package, count all the packages in the lot as 1, 2, 3,....., etc, up to *r* and so on where *r* is the integral part of  $N/n$ , *N* being the number of packages in the lot and *n* the number to be sampled. Every *r*th container thus counted shall be withdrawn to constitute the sample.

\*Method for random sampling.

## **B-2. PREPARATION OF SAMPLES**

**B-2.1** From each of the packages selected according to **B-1.2**, small portions of the material shall be taken from different parts so as to obtain a most representative sample of the package. The total quantity of the material taken from a package shall be at least three times the quantity needed for carrying out all the tests.

**B-2.2** The material obtained from each package in **B-2.1** shall be divided into three equal parts, each forming a test sample representing the package. One set of test samples each representing a selected package, shall be marked for the purchaser, another set for the supplier and the third set kept as a referee sample.

**B-2.3** All the test samples shall be immediately transferred to separate sample containers and shall be sealed air-tight and marked with full particulars necessary for proper identification, such as name of the supplier, place and date of packing, lot number, date of sampling, name of sample, etc.

**B-2.4** The referee sample consisting of a set of test samples shall bear the seal of both the supplier and the purchaser and shall be kept at a place till such time and under conditions as agreed to between the two for use in case of dispute.

## **B-3. NUMBER OF TESTS AND CRITERION FOR CONFORMITY**

**B-3.1 Number of Tests** — Each test sample in the set shall be tested individually for all the requirements of this specification.

**B-3.2 Criteria for Conformity** — The lot shall be declared to conform to the requirements of this specification if each test sample passes all the tests.

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